

**PATENT APPLICATION**  
**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re application of

Docket No: Q78557

Setsuo MISHIMA, et al.

Appln. No.: 10/715,568

Group Art Unit: 1742

Confirmation No.: 5060

Examiner: Kathleen A. McNelis

Filed: November 19, 2003

For: MARAGING STEEL AND METHOD OF PRODUCING THE SAME

**DECLARATION UNDER 37 C.F.R. § 1.132**

Mail Stop Amendment  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

I, Setsuo Mishima, hereby declare and state as follows:

In March of 1981, I completed the master course of steel metallurgy in the faculty of Engineering at Kyushu University.

In 1981, I entered the employ of Hitachi Metals, Ltd.

In 1985, I studied abroad at the Massachusetts Institute of Technology, Department of Materials Science and Engineering.

In 1987, I became a staff member in the steel making division at the Yasugi Works of Hitachi Metals, Ltd.

In 1995, I was transferred to Hitachi Metals Europe, Ltd.

In 1996, I returned to Japan and became a Senior staff member in the steel making division at the Yasugi Works of Hitachi Metals, Ltd.

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In 1998, I was posted to the Metallurgy Laboratory of Hitachi Metals, Ltd.

In 2003, I became a Primary engineer of the steel making division at the Yasugi Works of Hitachi Metals Ltd., and to date I still hold that position.

I have authored or co-authored the following research papers:

- 1) The Modeling of Fluid Flow and Heat Transfer in Mold Filling;
- 2) Behavior of Transformation of Calcined Dolomite to  $\text{Fe}_x\text{O}$ -CaO-SiO<sub>2</sub> type slag;
- 3) Mutual Diffusion Coefficient of FeO-MnO in molten slag of FeO-MnO-SiO<sub>2</sub>.

#### **Patents**

I, am the named inventor or am a named co-inventor in U.S. Patent No. 6,451,087 and three other U.S. Patents, four European Patents and 51 Japanese Patents.

My research and development work at Hitachi Metals, Ltd., can be characterized as follows:

- a. Improvement of die steel in machineability by controlling a sulfide form;
- b. Development and realization of a horizontal continuous casting technique for high alloy tool steels;
- c. Development of a refining technique for a high purity shadow mask material;
- d. Development of a manufacturing technique for a super high purity maraging steel;
- e. Study of the behavior of nonmetallic inclusions in the VAR process;
- f. Research and development of a method of evaluating nonmetallic inclusions by means of an acid extraction method.

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I have reviewed the specification and claims of the present application, the claims as amended in response to the official Action of 09/14/2007, have reviewed all rejections in this application, focusing primarily on the rejection of 09/14/2007, and have reviewed all prior art relied upon by the Examiner, including, but not limited to, JP 2001-214,212, which I refer to herein as JP '212.

I have performed, or have had performed under my direction and control, certain experimentation to establish the unexpected results achieved in accordance with the present invention, which experimentation is set forth below with reference to JP '212.

### **EXPERIMENTAL ROUTINE**

#### **JP '212**

JP '212 discloses producing a maraging steel from virgin raw materials by vacuum induction melting (VIM) and subsequent vacuum arc melting (VAR).

I thus prepared a maraging steel from virgin raw materials to correspond to the maraging steel disclosed in JP '212 in order to compare the JP '212 maraging steel to the maraging steel of the present invention.

#### **Raw Materials Prepared for the Experimentation**

- (1) electrolytic nickel (for the Ni component)
- (2) electrolytic cobalt (for the Co component)
- (3) metallic molybdenum (for the Mo component)
- (4) metallic titanium (for the Ti component)
- (5) metallic aluminum (for the Al component)

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(6) electrolytic iron (for the Fe component)

(7) an Ni-Mg alloy (for the Mg component)

\* All of these were virgin materials not containing scrap materials.

#### **Electrode Preparation**

Electrodes were prepared as follows.

(1) Two specimen steels, (No. 1 and No. 2) which corresponded to the maraging steel disclosed in JP '212 were melted by VIM using the above raw materials to prepare electrodes whose chemical compositions are given in Table 1. The melting involved 455 minutes.

In this regard, I understand the invention of JP '212 to be directed to a method of producing a Ti-containing steel. However, in claims 1 to 3 of JP '212, no chemical composition is recited for the steel.

In my opinion, specimen steels No. 1 and No. 2 are representative and conform to the example steel shown in JP '212 with respect to chemical compositions. See especially paragraph [0012] of JP '212.

Two specimen steels identified as No. 3 and No. 4 were prepared as maraging steels in accordance with the present invention. These two specimens were melted by VIM using the above raw materials to prepare electrodes. The chemical compositions are also given in Table 1. Again, the melting process involved 455 minutes.

I then measured the amount of Mg in specimen steel No. 1 and No. 2 in the electrodes. The measured Mg in the amounts of 3 to 4 ppm were derived from a lining wall which was made of magnesia in the melting furnace for VIM. Mg is an unavoidable impurity in the steel.

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Table 1

(mass%)												
Specimen Type	Form	C	Ni	Co	Mo	Ti	Al	Si	Mn	(N)	[O]	[Mg]
No.1	Elec-trode	.005	18.6	9.27	4.96	.47	.11	.05	.01	3	7	no addition 3
	Ingot	.005	18.6	9.29	4.99	.49	.13	.05	.01	3	2	1
No.2	Elec-trode	.004	18.5	9.27	5.01	.46	.12	.05	.01	4	7	no addition 4
	Ingot	.003	18.5	9.25	5.03	.48	.12	.04	.01	4	5	1
No.3	Elec-trode	.003	18.6	9.38	5.01	.45	.13	.01	.01	7	3	13
	Ingot	.003	18.6	9.38	5.05	.47	.14	.01	.01	4	3	2
No.4	Elec-trode	.003	18.6	9.26	5.00	.45	.12	.01	.01	4	3	10
	Ingot	.003	18.6	9.23	5.03	.48	.13	.01	.01	3	2	2

\*Note 1: The balance is Fe and unavoidable impurities.

\*Note 2: The amounts of nitrogen [N], oxygen [O] and Mg [Mg] are shown by "ppm" unit.

#### Preparation of Ingots

The electrodes made of specimen steels Nos. 1, 2, 3 and 4 were subjected to VAR to obtain ingots. Each ingot had a weight of 1 ton. The chemical composition of each ingot is also shown in Table 1.

The VAR processing was conducted at: a melting rate of 210 Kg/Hr under a vacuum of 0.002 Torr using a mold having an inner diameter of 350 mm.

#### Slab Preparation

The ingots obtained were subjected to hot forging in a conventional manner to obtain slabs.

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#### **Obtaining Specimens from the Electrodes before VAR and from the Slubs after VAR**

Specimens were obtained from the electrodes and the slubs in order to make observe and measure (TiN) type nonmetallic conclusions. Each specimen weighed ~~10~~<sup>5</sup> grams.

*S.M.  
Feb. 13. 2008*

#### **Extraction of Nitrides and Carbonitrides from the Specimen Steels**

Each specimen steel having a weight of ~~10~~<sup>5</sup> grams was dissolved using a mixed acid and the resulting dissolved products subjected to filtration to obtain the nitrides and carbonitrides from each specimen steel.

*S.M.  
Feb. 13. 2008*

#### **Size Determination of Nitrides**

For each specimen, the thus obtained nonmetallic inclusions were precipitated and observed using a scanning electron microscope (SEM).

In the nitrides observed by SEM, the maximum size of nitride type nonmetallic inclusions was determined for each specimen. The thus determined sizes are given in Table 2.

The following should be noted:

Since TiN is a hard material, its size is not changed by hot forging the ingots.

The sampling positions of the specimen steels from the electrodes and the slubs were predetermined and were generally in the same position in each electrode and in each slub.

In the size determination of the nitrides, since the nitrides are rectangular, a long side (a) and a short side (b) were both measured to obtain an area of "a x b", whereafter a circle having the same area as the rectangular area was determined and, further, the diameter of the circle was determined, with the diameter of the circle being regarded as representing the diameter as a maximum size of the nitride.

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The results are shown in Table 2.

TABLE 2

Specimen Type	Maximum size of nitride type nonmetallic inclusions	
	Electrode (before VAR)	Slab (after VAR)
No. 1	4.8 $\mu\text{m}$	6.6 $\mu\text{m}$
No. 2	4.2 $\mu\text{m}$	5.9 $\mu\text{m}$
No. 3	4.8 $\mu\text{m}$	4.4 $\mu\text{m}$
No. 4	4.1 $\mu\text{m}$	3.6 $\mu\text{m}$

#### Evaluation of Experimental Results

From the results shown in Table 2, although the maraging steel specimens corresponding to JP '212 and the present invention were produced in using the same virgin raw materials, in the invention maraging steel specimens which had Mg added thereto, TiN was not coarsened even after VAR. In distinction, in the maraging steel specimens corresponding to JP '212 where there was no positive addition of Mg, TiN was coarsened after VAR by an amount of about 27 to 29%. In my opinion, this difference is due to the intentional addition of Mg in accordance with the present invention as opposed to the lack of positive or intentional addition of Mg in accordance with JP '212.

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I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: February 13, 2008

Setsuo Mishima  
Setsuo Mishima